Research Highlight #156

Broadband W-band rapid frequency sweep considerations for Fourier transform EPR

Robert A. Strangeway, Theodore G. Camenisch, Jason W. Sidabras, Richard R. Mett, James R. Anderson, Joseph J. Ratke, Witold K. Subczynski, James S. Hyde

National Biomedical EPR Center, Department of Biophysics, Medical College of Wisconsin

Introduction: A number of EPR spectrometer designs have been described for use at W-band.[1,2] Our group extended loop-gap resonator (LGR) technology to W-band in 2007,[3] which enabled development of the concept of W-band broadband EPR. The earliest paper in this field was published by our group in 2010,[4] and it provides a framework of ideas that underlie the advancements highlighted here. That paper established that it was feasible to invert the magnetization of a nitroxide radical spin label in fluid solution by rapid sweep of the microwave frequency across the spectrum, and that the signal could then be observed by detection of the free induction decay (FID). We believe this was the first EPR report of FID detection following sweep of the microwave frequency rather than magnetic field and it was concluded that feasibility for FT EPR by frequency sweep broadband W-band technology had been established.

Results: Our work highlighted here[5] builds on the technical advances of a new multi-arm W-band (94 GHz) EPR spectrometer highlighted in #152 in last year's RPPR. To review, the new design can be configured for continuous wave (CW) experiments, including field- and frequency-swept EPR, saturation-recovery EPR, and pulsed electron-electron double resonance (ELDOR) and utilizes a LGR. The superconducting magnet has a horizontal magnetic field and provides access through a side port. The bridge is mounted on a table that rests on a granite slab and is cleverly supported by air bearings and readily moved in a horizontal plane to introduce the sample through the side port of the magnet. The microwave power chain contains four microwave sources: microwave synthesizers in the 2 GHz range, two low-noise Gunn diode oscillators (33 and 51 GHz), and an yttrium iron garnet (YIG) oscillator (6.5 to 9.5 GHz). The synthesizers are time-locked and provide digital accuracy of frequency differences between arms; the in-house developed Gunn diode oscillator pair dominates the microwave phase noise, which is exceptionally low, and the YIG oscillator provides frequency agility. There are two sample-irradiation arms and a receiver arm. Path-length equalization, which occurs at the intermediate frequency of 59 GHz, is analyzed. A directional coupler is favored for separation of incident and reflected power between the bridge and the LGR. Microwave leakage of this coupler is analyzed. An oversize waveguide with hyperbolic-cosine tapers (see Highlight #155) couples the bridge to the LGR and results in greatly reduced microwave power loss.

Implications: The sensitivity of the instrumental setup shown in Figure 1, which was designed for free radical studies at room temperature, has been improved by an estimated factor of 10 relative to the previous published report.

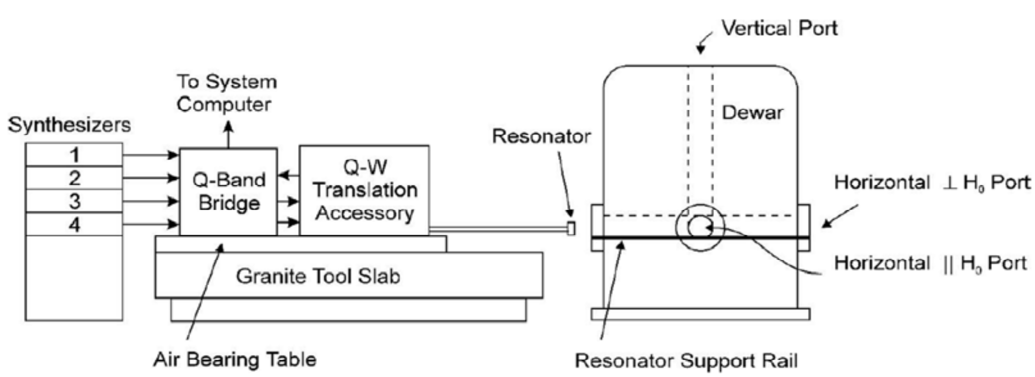

Figure 1. Overall assembly of the W-band spectrometer

[1] Grinberg OY, Berliner LJ (eds.). (2004) Very High Frequency (VHF) ESR/EPR. Biological Magnetic Resonance. New York: Springer Press.

[2] Misra SK (2011) Multifrequency Electron Paramagnetic Resonance: Theory and Applications, Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim, Germany.

[3] Sidabras JW, Mett RR, Froncisz W, Camenisch TG, Anderson JR, Hyde JS. (2007) Multipurpose EPR Loop-Gap Resonator and Cylindrical TE011 Cavity for Aqueous Samples at 94 GHz. Rev Sci Instrum. 2007. 78, 034701.

[4] Hyde JS, Strangeway RA, Camenisch TG, Ratke JJ, Froncisz W. (2010) W-band Frequency-Swept EPR. J Magn Reson. 2010. 205, 93-101. PMCID: PMC2885579

[5] Strangeway RA, Camenisch TG, Sidabras JS, Mett RR, Anderson JR, Ratke JJ, Subczynski WK, Hyde JS. (in submission) Broadband W-band Rapid Frequency Sweep Considerations for Fourier Transform EPR. [in submission to Cell Biochem Biophys]